U.S. DEPARTMENT OF COMMERCE
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PB-287 157

Evaluation of Cartridge Filters for Removal of Small Fibers from Drinking Water

Environmental Research Lab -Duluth, MN

Aug 78

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO.	2.	3. PB 287157
EPA-600/J-78-049	JOURNAL ARTICLE	

4. TITLE AND SUBTITLE	5. REPORT DATE
Evaluation of Cartridge Filters for Removal of Small Fibers from Drinking Water	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) P. M. Cook, G. E. Glass, D. R. Marklund, and J. H. Tucker	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Environmental Protection Agency Environmental Research Laboratory-Duluth 6201 Congdon Boulevard Duluth, Minnesota 55804	10. PROGRAM ELEMENT NO. 1BA608
	11. CONTRACT/GRANT NO.
12. SPONSORING AGENCY NAME AND ADDRESS Same as above	13. TYPE OF REPORT AND PERIOD COVERED
	14. SPONSORING AGENCY CODE EPA-600/03

15. SUPPLEMENTARY NOTES REFERENCE: Cook, P. M., G. E. Glass, D. R. Marklund, and J. H. Tucker. 1978. Evaluation of Cartridge Filters for Removal of Small Fibers from Drinking Water. J. Am. Water Works Assoc. 70(8):459-464.

16. ABSTRACT

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17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Asbestos Fibers Amphiboles Water supply Filtration X-ray diffraction Electron microscopy Turbidity Suspended sediment Particles		06 07 08 11 14

REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U.S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS *(This Report)*	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	8
	20. SECURITY CLASS *(This page)* UNCLASSIFIED	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

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P.M. Cook, G.E. Glass, D.R. Marklund, and J. H. Tucker

Prior to completion of new water treatment facilities in Duluth, Minn., contamination of the community's water supply by amphibole fibers led to widespread use of individual filtration systems in homes and public buildings. Laboratory tests of several types of cartridge filters, backed by extensive field monitoring of operating filter installations, revealed a number of performance problems and shed light on how to correct them.

Many different microfiltration devices are in use today for the removal of fine particles from water. In recent years great interest has developed in the filtration of drinking water to remove small asbestos fibers. Curiously, the first reports of asbestos-fiber contamination of fluids concerned fibers in beverages[1] and parenteral drugs,[2] as a result of filtration through asbestos-fiber filters.

Asbestos fibers are known to cause cancer in man following exposure by inhalation, but the health risk of ingesting fibers is only suggested by the approximately threefold excess of gastrointestinal cancers often observed for asbestos workers,[3] who presumably swallow many fibers that are first inhaled and then cleared from the lung by ciliary action. Intraperitoneal injections of asbestos fibers have produced malignant neoplasms in animals.[4] No long-term epidemiological study of a

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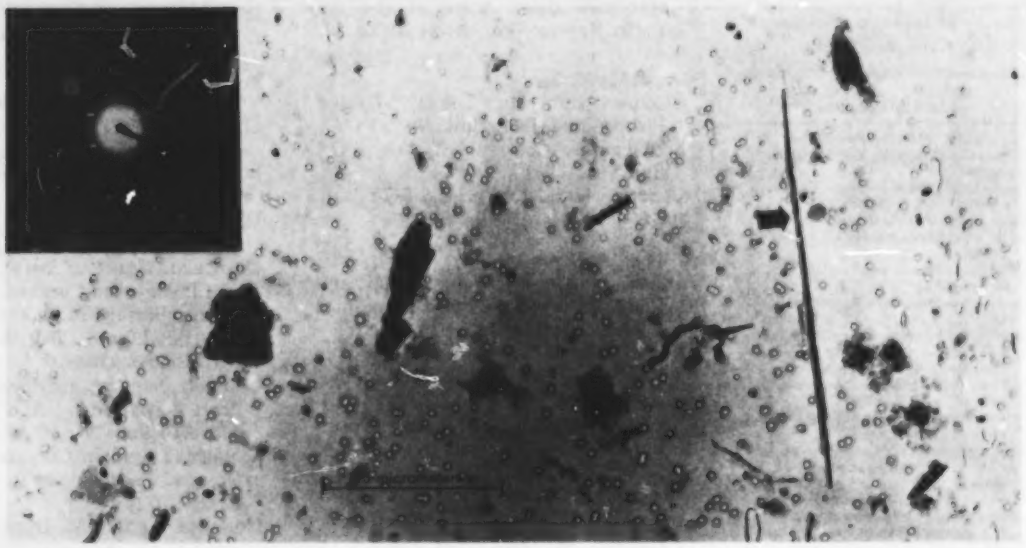

Fig. 1. Electron micrograph of particles in Duluth tap water shows amphibole fibers, other mineral particles, diatom fragments, and organic debris. Amphibole fibers (arrow at right) can be identified by selected area electron diffraction patterns (insert) observed on the electron microscope. The area shown represents 0.000014 ml of water.

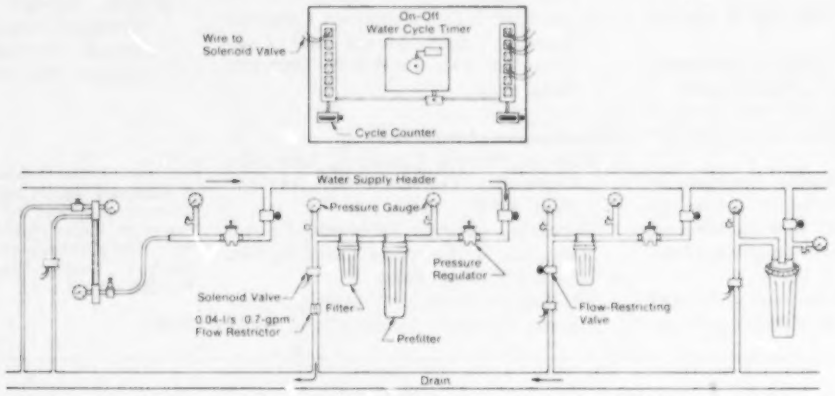

Fig. 2. Typical testing arrangement for cartridge filter tests performed with 30-s on–30-s off cycled water flow to simulate actual filter use conditions.

human population exposed only by ingestion to large concentrations of asbestos or other mineral fibers has been reported. However, excess gastrointestinal cancer mortality[5] or morbidity[6] has not been detected among residents of Duluth, Minn., after nearly eighteen years of ingesting water contaminated with amphibole fibers.* Surveillance of the Duluth population will continue since long time lapses are known to exist between initial exposure to asbestos fibers and diagnosis of asbestos-caused cancers.

The US Environmental Protection Agency (EPA) announced in June 1973 that the Duluth, Minn., water supply was contaminated with amphibole mineral fibers. The source of these fibers is the 60 000 tonnes (67 000 tons) per day of taconite (iron ore) tailings that are discharged into Lake Superior at Silver Bay, Minn.[7] Although this discharge began in October 1955, the Duluth water supply until recently used Lake Superior water without filtration. A filtration plant that employs flocculation with a mixed granular media filtration system has been operational since January 1977.

Since the discovery of fiber contamination of the drinking water, the EPA Environmental Research Laboratory-Duluth has tested twelve different types of cartridge filters to determine which will remove inorganic fibers continuously without adding fine particles to the filtered water through filter-media migration. The throughput and cost data collected during most of these tests have been evaluated by other agencies, which purchased filters for installation in the Duluth area as an interim measure for providing fiber-free water to the public. Most filter testing was conducted in May–June 1974[8] and February–April 1976 at the request of the US Army Corps of Engineers or, for the monitoring of operational cartridge filter installations, at the request of the City of Duluth. The US Army Corps of Engineers was designated by the US District Court (United States et al vs Reserve

*Amphibole minerals are members of a class of hydrated silicates that share a common crystal structure. These minerals can crystallize during rock formation in an asbestiform habit (bundles of individual fibers). Asbestiform amphibole minerals used commercially as asbestos are amosite, crocidolite, anthophyllite, tremolite, and actinolite. A fiber in a water sample is defined as any particle that is observed to have a length-to-width ratio exceeding or equal to 3.0. Some nonasbestiform amphibole mineral crystals, when crushed, form cleavage fragments that are microscopically very similar, or identical, to small fibers that result from the crushing of asbestiform amphibole rock. Approximately 95 per cent of the asbestos used in North America is chrysotile, a fibrous serpentine mineral with a distinct tubular structure.

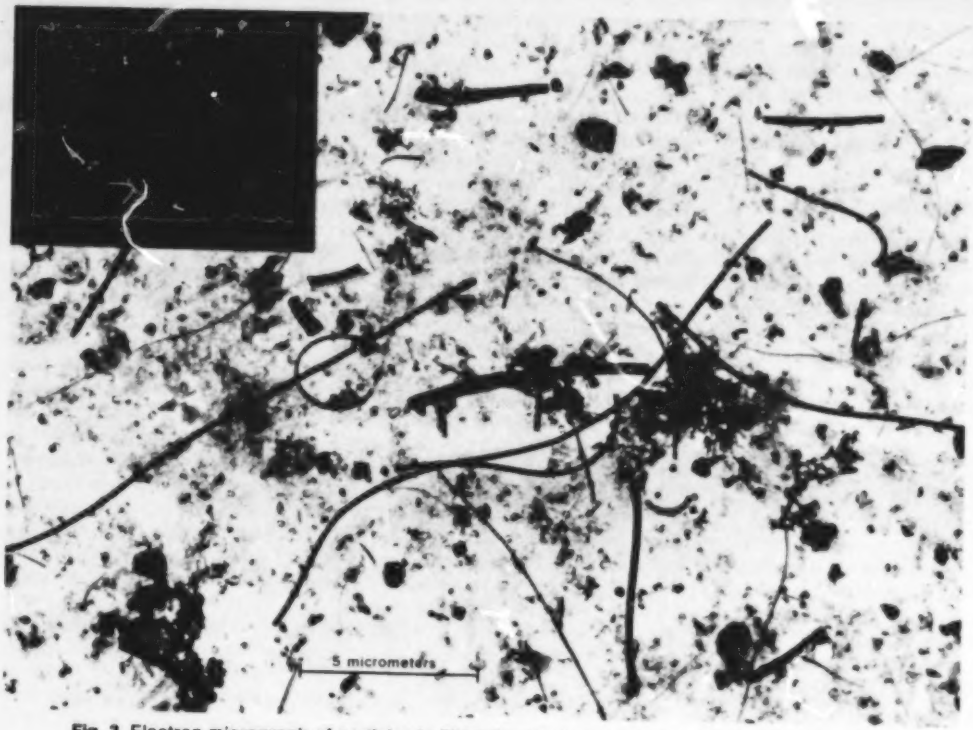

Fig. 3. Electron micrograph of particles in filtered water from a school drinking fountain. Most are glass fibers that migrated from the prefilter through breaks in the filter membrane. Glass fibers are amorphous (no SAED) and produce energy dispersive x-ray spectra with a silicon peak at 1.74 keV (insert). The area shown represents 0.00017 ml of water.

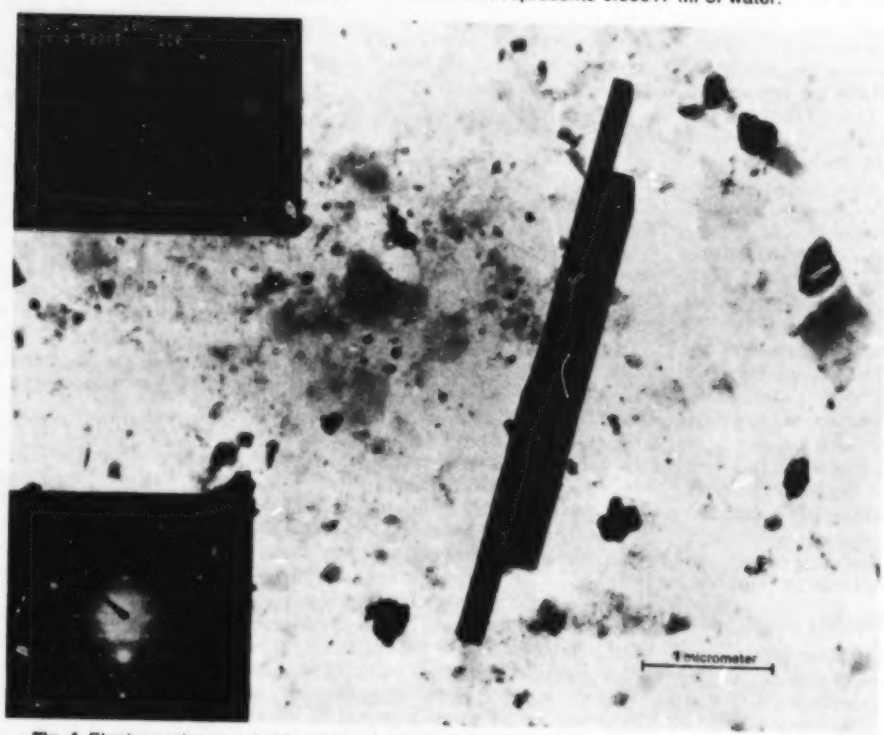

Fig. 4. Electron micrograph of particles in filtered water from the laboratory test of a depth-type filter constructed with titanate fibers. Filter media migration resulted in fibers in the filtered water such as that shown, which produced distinctive SAED patterns (lower insert) and energy dispersive x-ray spectra with a titanium peak at 4.50 keV (upper insert).

Mining Co. et al) as the agency responsible for providing filters at fire halls, schools, churches, restaurants, and public buildings. Approximately 1650 filters have been installed in the Duluth area under this program. An unknown number of filters have been purchased privately for use in homes.

Laboratory Test Water

Duluth drinking water was used in all tests to determine fiber-removal efficiency. The average concentration of amphibole fibers that can be positively identified by electron microscope examination of Duluth water samples is approximately 120 million fibers per litre. Fiber concentrations vary with seasonal and weather effects on Lake Superior circulation.[9]

A typical electron microscope field of view of the test water contains several amphibole fibers (Fig. 1). They can be positively identified by electron diffraction patterns (insert, Fig. 1), which are obtained rapidly on most modern electron microscopes. The amphibole fibers have a mean length of about 1.6 μm and a mean width of about 0.2 μm. The size distribution of amphibole fibers identified is somewhat artificial since many fibers are too small and a few are too large to allow identification by electron diffraction. Total fiber concentrations measured are typically more than double the amphibole fiber concentrations because of the presence of many "ambiguous" fibers that cannot be positively identified as amphibole or nonamphibole.[9] Ambiguous fibers have mean lengths of approximately 1.0 μm and mean diameters of 0.1 μm.

Concentrations of total suspended solids in Duluth water average approximately 1 mg/l but during storms can exceed 50 mg/l. The average amphibole mineral-mass concentration is 0.20 mg/l. Particle-size distributions during storm-caused turbid water conditions are skewed to larger sizes because of the presence of freshly stirred-up lake bottom sediments in the water. Thus measurements of filtration efficiency are complicated by periodic changes in particle-size distribution as well as total solids loading. Water temperature is less than 5 C except during July–September, when it occasionally exceeds 10 C. Unregulated water-line pressure was about 420 kPa (60 psi) during the tests.

Laboratory Test Facility

The filter-testing arrangement used during 1976 is shown in Fig. 2. The 1976 tests differed from previous (1974) tests performed by the US Army Corps of Engineers[8] in cooperation with the USEPA primarily in the use of timer-activated solenoid valves to create approximately 30-s on–30-s off cycles of water flow through the filters. This test procedure simulated actual use conditions observed in Duluth rather than the continuous-flow conditions used in earlier tests. In most tests the complete filter life cycle was tested—i.e., from start to point of obvious failure to remove fibers or point of severely restricted flow.

Each filter manufacturer was asked in 1976 to specify conditions necessary for proper use of his filter. These conditions should be observed for installation in Duluth buildings. Most filters were thus tested with pressure regulators or flow regulators, or both, installed to produce optimum filter performance. One filter was tested in 1976 with and without a 5-μm prefilter. In 1974 filters were tested at flow rates restricted to either 0.04 l/s (0.7 gpm) for single cartridge filters or 0.2 l/s (4.0 gpm) for multiple cartridge filter arrangements.[8]

Pressure gauges were installed so that raw water and filtered water pressures could be recorded frequently. In the 1976 tests the downstream pressure gauges did not give pressure-drop information because the flow from the filter was normally less than the flow allowed by the flow regulator downstream. A pressure gauge was installed on a few filter housings in a position that allowed measurement of pressure drop across the filter membrane.

Flow rates were determined simply by measuring the volume of filtered water produced over a stopwatch-determined period of time. Electrically operated counters recorded the cumulative number of 30-s on–30-s off cycles.

Analysis of Water Samples

Water was normally cycled through the filters continuously until completion of the tests except for shutdown on weekends. Turbidity of raw and filtered water samples was measured by turbidimeter* one to three times per day. The linear correlations for 1976 test data between turbidity (T in nephelometric turbidity units ntu) and total suspended solids (S in mg/l) are expressed by the equations $T = 1.24S - 0.01$ for filtered Duluth drinking water and $T = 0.78S + 0.23$ for unfiltered Duluth drinking water, with correlation coefficients of 0.97 ($N = 28$) and 0.97 ($N = 16$) respectively. The different slopes for these two strong correlations probably result from different particle-size distributions in filtered water (smaller particles) and unfiltered water (larger as well as more particles) and thus decreased scattering of light per unit mass of particles in unfiltered water as compared to filtered water. Even if turbidity is assumed to be exactly equal to total suspended solids ($T = S$) for all water samples, turbidity measurements provide a fast, convenient, and reliable check of suspended solids removal. Turbidity, however, is not normally a sensitive enough measure to indicate when filter-media migration to finished water has occurred.

X-ray diffraction methods[10] were used to determine amphibole mineral-mass concentrations. Although this technique does not directly measure the concentration of amphibole fibers in the water, previously established correlations[9] indicate that a reliable estimate can be made. The concentration of total suspended solids is determined gravimetrically as part of the x-ray diffraction analysis, which is less expensive and time consuming than the rigorous electron microscope determination of fiber concentration. Since large volumes of filter effluent water (up to 100 l) are filtered for x-ray diffraction analysis, an integrated measurement over many on–off cycles is obtained, whereas turbidity and electron microscope fiber counts are essentially instantaneous measurements because of the small volume of water collected for analysis.

The basic, rigorous analytical technique for determining fiber concentrations in water is transmission electron microscopy. Sample preparation and analysis for 1974 tests were performed by the Dow Chemical Company.[8,11] The technique employed by the Environmental Research Laboratory–Duluth for 1975 and 1976 tests was slightly different, but the results produced are comparable.[9] Both techniques involve filtration of the water sample on a small membrane filter and direct transfer of the sample from the membrane to an electron microscope grid, which is systematically examined on the transmission electron microscope. This technique is particularly important for obtaining fiber-size information and for determining filter-media migration problems. Individual particles can be identified by morphology; crystal structure, as determined by selected area electron diffraction (SAED); and elemental composition, as determined by energy dispersive x-ray spectroscopy (EDS).

Filter Types Tested

Laboratory cartridge-filter tests were performed, either in 1974 or 1976, on four different depth-type filters constructed of glass or some other inorganic fibers; five different membrane filters with membranes constructed of polymers such as polycarbonate, polyvinyl chloride, or cellulose ester; two hollow fiber membrane filters; and a diatomaceous earth–activated carbon filter. Some of the membrane filter cartridges also contained prefilters constructed from glass fibers. Other fiber-removal techniques tested since 1973 included distillation, magnetic separation, reverse osmosis, and conventional water supply treatment techniques such as sand filtration, diato-

*Model 2100A, Hach Chemical Co., Ames, Iowa

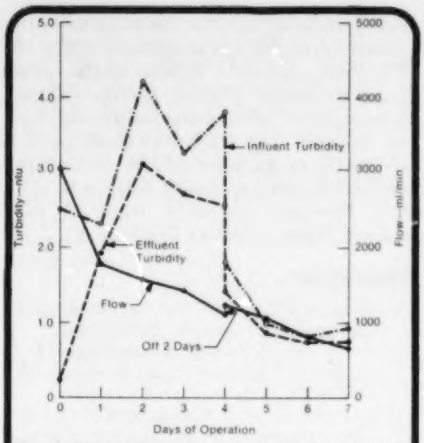

Fig. 5. Filter efficiency as measured by turbidity of filtered and unfiltered water over the test period for a membrane filter that failed during the first day of cycled flow operation. Decreasing flow rate during the test is also plotted.

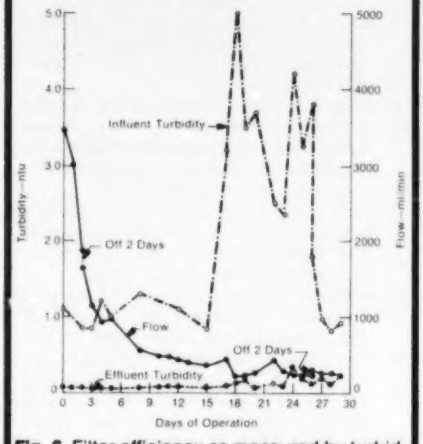

Fig. 6. Filter efficiency as measured by turbidity of filtered and unfiltered water over a 28-day test period of cycled flow operation for a diatomaceous earth–activated carbon cartridge filter. Flow rate during the test is also plotted.

maceous earth filtration, and granular filtration following flocculation.[12]

Monitoring Filters in Use

Filters, when installed in fountains, homes, schools, and so forth to produce fiber-free water for drinking and cooking, do not necessarily perform as well as laboratory tests indicate they should. Some of the possible problems are improper installation, leaky seals around cartridges, excessive water pressure, water-hammer effects, and excessive water-flow demand on the filter. The Environmental Research Laboratory–Duluth has tested hundreds of raw and filtered water samples for the City of Duluth and has used x-ray diffraction and occasional electron microscope results to supplement an extensive monitoring program based on turbidity testing of filtered water.

Filters were initially installed with the assumption that they should be replaced when "plugged." In late March 1975 a severe northeast storm caused resuspension of so much lake sediment that suspended solids in Duluth drinking water exceeded 25 mg/l for several days. A check of eleven membrane-type multiple cartridge filter installations indicated that seven were passing unsatisfactory concentrations of amphibole fibers into the finished water rather than plugging up. The filter manufacturer recommended more frequent cartridge changes and the installation of accumulator tanks to soften water-hammer effects. Continual monitoring of these filters indicated that some processed water had high concentrations of suspended solids and amphibole fibers because of water bypass, which was caused by faulty sealing of filters in multiple cartridge units.

In October 1975 a water sample from a 10-in. cartridge filter of the type installed throughout Duluth (pleated membrane with fiber glass prefilter) was examined by electron microscopy. Although this filter had provided drinking water for more than one year, turbidity analysis indicated satisfactory performance. The sample, however, contained, in addition to amphibole fibers, approximately 90 million glass fibers per litre of water. These glass fibers could only have resulted from the loss of prefilter material that passed through cracks in the membrane, since glass fibers are virtually undetectable in all unfiltered water samples examined. Twelve more water samples from filters installed in homes, schools, and restaurants were inspected by electron microscopy. Five samples, four from filters which had been in operation for more than one year, contained very high fiber glass concentrations. A sample from a school filter (Fig. 3), for example, contained approximately 530 million fiber glass fibers per litre. At least two samples contained concentrations that exceeded a billion fibers per litre. Subsequent turbidity testing of many filters by the City of Duluth led to the replacement of hundreds of filter cartridges and a more intensive monitoring program. One hundred and forty of 168 filters in schools were producing filtered water with unsatisfactory turbidity levels. Many of these filters had been in operation for more than one year. Laboratory tests and observation of filter failures revealed that intermittent use at line pressures often exceeding 350 kPa (50 psi) caused a flexing of the pleated membrane such that the pleat edges eventually cracked under water-hammer pressure surges.

Filter Testing Results

The monitoring during 1975 of operational pleated membrane cartridge filters in the Duluth area resulted in the following observations that were not available from laboratory filter testing:

1. Continuous-flow laboratory testing of filters did not model actual intermittent-use conditions in Duluth.
2. Cartridge filters should only be installed and operated under conditions carefully determined by the manufacturer for fail-safe performance over the period of use.
3. The point at which cartridge replacement is necessary should be clearly defined by the manufacturer and identifiable by the user.
4. The use of any inorganic fiber as prefilter material for a membrane filter is likely to result in fiber contamination of the filtered water as soon as flaws or cracks develop in the membrane.
5. Even the strongest membrane material, when pleated, will weaken at the pleat ends with intermittent use, especially as pressure drop across the membrane increases under unregulated line-pressure conditions.
6. Watertight seals must be obtained for filter cartridges in their housings to prevent water bypass.

Subsequent laboratory testing of filters was performed with the water on-off cycle system previously described. Testing in November–December 1975 led to the installation in Duluth of pleated membrane filters with no fiber glass prefilter. The membrane area was doubled and a second, larger pore membrane was wrapped about the core of the filter so that membrane pleat breakage would result in the rapid plugging of the core-wrap membrane. Optimum pore size of the membrane filter for Duluth water was found to be slightly greater than 0.2 μm. These filters were installed in Duluth, many with line pressures regulated to 140 kPa (20 psi), and appear to produce low amphibole fiber concentrations over the life of the filter with no risk of glass-fiber contamination. Filters are replaced when either low flow or slightly elevated filtered water turbidity is observed during frequent tests.

Although the pleated membrane filters installed in Duluth gave no evidence of glass-fiber loss during laboratory tests, the inorganic fiber depth-type filters appeared to lose fibers in varying amounts throughout the laboratory test periods. Highest concentrations were observed under intermittent-flow conditions or after pressure-drop buildup across the filter. These filters probably release the largest concentrations of fibers with the pressure surge that occurs when the water is turned on during intermittent use. Total fiber concentrations of four filtered water samples randomly selected from four separate tests of the same fiberglass filter were 90, 380, 440, and 270 million total fibers per

litre. Most of these fibers were identifiable as glass, with more amphibole fibers present in the samples collected toward the end of filter tests. Synthetic fibers such as glass have been shown to produce malignant neoplasms after implantation in rat tissue. Because carcinogenicity of fibers has been concluded by some investigators[13] to depend on durability and dimension rather than physicochemical properties, durable synthetic fibers were regarded in these tests as highly undesirable contaminants.

Another type of fiber filter produced under continuous-flow conditions filtered water that contained titanate fibers (Fig. 4). Some of these fibers were large enough to be observed without the aid of a microscope, but most were similar in size to the fiber pictured. These fibers produced electron diffraction patterns (lower left insert, Fig. 4) similar to amphibole fibers but with a distinctively different layer line spacing. Some titanates have the same C2/m-monoclinic space group symmetry as do amphibole minerals, but smaller cell parameters, so the SAED similarity is not surprising. The upper left insert of Fig. 4 is an energy dispersive x-ray spectrum produced by the fiber. This spectrum contains a titanium peak at 4.50 keV.

The end point of these tests was often near total breakthrough of a membrane such as shown in Fig. 5. Under on-off-cycled flow conditions this filter lost all filtration ability within a day as filtered water turbidity rapidly became roughly equal to raw water turbidity.

A more favorable test result is demonstrated in Fig. 6. This diatomaceous earth-activated carbon cartridge filter was run at 420 kPa (60 psi) line pressure for 30 days under on-off-cycle flow conditions. An exponential drop in flow occurred, and very low filtered water turbidity and amphibole concentrations were measured over the first eighteen days of operation. After that, filtered water turbidity increased erratically to a high of 0.25 ntu, but amphibole concentration remained low (0.005 mg/l on day 27) despite very high raw water suspended solids and amphibole concentrations during the later days of operation. Some activated carbon particulate appeared in filtered water samples collected at the end of the test, and the amphibole concentration rose to 0.02 mg/l (compared to 0.36 mg/l in unfiltered water). Several filtered water samples examined by electron microscopy did not have detectable concentrations of amphibole or other mineral fibers (< one million fibers per litre). This filter was tested well beyond the point at which the cartridge should be replaced because of reduced flow. The filter did not operate to remove a constant percentage of the suspended solids present. A fourfold increase in raw water suspended solids did not increase filtered water suspended solids or amphibole concentrations.

Figures 5 and 6 are provided only as examples of laboratory filter-test results. Almost every test run was unique because of differences in filter design and the changes in filter performance taking place over its period of use.

Summary

The purpose of filter testing in Duluth was to determine which filters would consistently remove at least 95 per cent of the amphibole fibers from Duluth drinking water without adding other contaminants such as glass fibers or objectionable chemicals. Very few tests produced this result. Most filters are designed to remove all particles above some nominal or absolute size and perform this function well early in the period of their use. Often, however, a steady or dramatic drop in filtration efficiency was observed during testing. Filter-media migration was also a common problem. Measurements of decrease in pressure or flow rate do not often serve as reliable indicators of unacceptable water quality. Certainly, where the primary interest is the removal of a particular type or size of particle, the particle content of the filtered water must be measured frequently to establish the performance characteristics of the filter under a particular set of operating conditions.

Field testing of operating filters was instrumental in the development of filtration systems that could reliably provide fiber-free water in Duluth. The user, particularly if not expert in filtration technology, should receive the following information from the filter manufacturer before purchasing filtration equipment:

1. Specific recommended operating conditions for the particular application of the filter; e.g., optimum and maximum line pressure, optimum and maximum flow rate, proper installation and maintenance procedures, and restriction to continuous-flow operation

2. A dependable criterion for determining the useful life of the filter so filter elements can be changed before the filtered water quality becomes unacceptable

3. A measure of the probability and consequences of failure before filter element replacement (e.g., 1 per cent chance of membrane rupture, which would release accumulated particles plus prefilter material)

4. Loss of filtration efficiency to be expected for the particle size of interest over the projected useful filter life.

Acknowledgements

The authors are indebted to G.R. Endicott of the Environmental Research Laboratory-Duluth for his assistance in constructing filter test apparatus and to P.L. Smith and D.G. Wilson of the Lake Superior Basin Studies Center of the University of Minnesota-Duluth for water analysis. The work was made possible by the cooperation of H.C. Anderson of the US Army Corps of Engineers and D.L. Peterson and T.M. Biele of the Duluth Water and Gas Department.

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